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FINAL REPORT

FOR

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Design Synthesis and Characterization of Novel Polydiacetylenes Using New Analytical Technique

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REPORT DOCUMENTATION PAGE

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Final Report under Contract N00014-87-K-0131

12. PERSONAL AUTHOR(S)
Sukant Tripathy

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16. SUPPLEMENTARY NOTATION

17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
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19. ABSTRACT (Continue on reverse if necessary and identify by block number)

During the project period significant progress were made in the design, synthesis and analysis of a number of electroactive polymers. Polymers were assessed for their structural features, electroni and optical properties and their application in molecular electronic devices. Substantial progress during the project period has led to expansion of research activities into a number of different areas including conducting monolayers, 3rd order nonlinear opitcal polymers and newly designed materials with second and 3rd order nonlinear opitcal properties.

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DD Form 1473, JUN 86 *Previous editions are obsolete.* SECURITY CLASSIFICATION OF THIS PAGE

Final Technical Report

The principal activities during this $2^1/_2$ year funded project may be devised into three categories.

1. Design and synthesis of novel electroactive macromolecular systems derived from functional monomers.

2. Preparation of ultrathin film in the forms of mono and multilayers, thin film single crystals and spun on films.

3. Investigation of molecular order, electronic and optical properties and establishment of structure property relationships.

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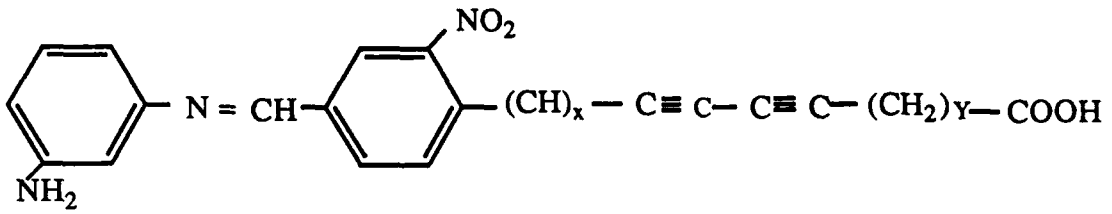
1. Monomers of the general structure.



were synthesized.

These monomers are synthesized where a combination of several other donor and acceptor groups can replace the amino and nitro groups. (Report No. 4)

These monomers were then organized into monolayer form and polymerized via the diacytylene functionality. These monolayers are expected to have novel linear and nonlinear optical properties; large second order co-efficient due to the noncentrosymmetric alignment of the side group and a large third order coefficient due to the polydiacetylene backbone. In addition novel interplay of the properties; side group induced effects in the backbone were anticipated.

2. Monolayers of these electrooptic materials with diacetylene functionality were prepared (technical report No. 6, 1990), In addition polypyrrole was prepared as a monolayer by solubilizing in a mixed solvent system. Conducting monolayers and multilayers of polypyrrole and its analogs were prepared using a L-B film balance. Films were transferred to appropriate substrates and were picked up on microscope grids for electron microscopy studies among a number of other investigations. Scanning electron microscopy revealed a smooth coverage over a large area and electron diffraction studies established a different crystalline structure to those observed in the films synthesized by the standared electrochemical techniques. Mixed monolayers of pyrrole with hexadecyl pyrrole was also synthesized. These were polymerized using $FeCl_3$ in the subphase (Report no. 3)

3. Optical electronic and structural properties of this and other electroactive materials were investigated.

The optical properties of partially hydrogenated polyacetylene chains were investigated using quantum chemical techniques (Report no. 1). Order in the L-B film of hexadecyl pyrrole was studied using near edge X-ray fine structure spectroscopy using the Brookhaven synchrotron light source (report 2). Grazing incident FT-IR spectroscopy was carried out to establish structural order in these monolayers.

Optical properties of polydiacetylenes with different side groups was assessed (Report 5) significant spectral shifts in the backbone absorption spectrum were anticipated as electroactive side groups were attached to the diacetylene backbone.

Two review articles were prepared during the course of this research. "Nonlinear Optics and Organic Materials" was published in two parts in Chemtech (Report 7) and a chapter in "Optical Spectroscopy of Polymers" H. Bassler Ed. Elsevier 1990 was published with the title "Nonlinear Spectroscopy of Polymers".

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PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT ACCESSION NO.

11. TITLE (Include Security Classification)

Polyheterocycle Langmuir-Blodgett Films

12. PERSONAL AUTHOR(S) X.Q. Yang, J. Chen, P.D. Hale, T. Inagaki, T.A. Skotheim, D.A. Fischer, Y. Okamoto, L. Samuelson, S. Tripathy, K. Hong, I. Watanabe, M.F. Rubner and M.L. denBoer

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15. PAGE COUNT
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16. SUPPLEMENTARY NOTATION

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17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
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19. ABSTRACT (Continue on reverse if necessary and identify by block number)

Two types of polyheterocycle Langmuir-Blodgett films have been fabricated: (1) copolymers of pyrrole and 3-alkyl pyrrole (3-hexadecylpyrrole and octadecylpyrrole) LB films; (2) mixtures of poly(3-alkyl thiophene) and stearic acid LB films. The orientation of single- and multi-layer films on platinum substrates have been studied by Near Edge X-ray Absorption Fine Structure (NEXAFS) spectroscopy which also provides information about interaction between the aromatic groups and the metallic substrate. The alkyl substituted pyrrole monomers form highly ordered multi-layer LB films with the hydrocarbon chains perpendicular to the substrate, while the LB films of copolymers of pyrrole and alkyl substituted pyrrole are more disordered. In the case of mixtures of poly(3-alkyl thiophene) and stearic acid LB films, the hydrocarbon chains of the stearic acid molecules are highly ordered. The poly(3-alkyl thiophene) components, on the other hand, exhibit random orientation of the thiophene moieties. The orientation of the hydrocarbon chain of the poly(3-alkyl thiophene) varies with the chain length, from random to highly ordered along the surface normal of the substrate, as the chain length changes from 4 carbon units to 18 carbon units.

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PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT ACCESSION NO.

11. TITLE (Include Security Classification): Highly Ordered Thin Films of Polyheterocycles: A Synchrotron Radiation Study of Polypyrrole and Polythiophene Langmuir-Blodgett Films

12. PERSONAL AUTHOR(S): T.A. Skotheim, X.Q. Yang, J. Chen, P.D. Hale, T. Inagaki, L. Samuelson, S. Tripathy, K. Hong, M.F. Rubner, M.L. denBoer and Y. Okamoto

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15. PAGE COUNT
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SUPPLEMENTARY NOTATION: Synthetic Metals 28 C229-C236 (1989), Proceedings of ICSM conference, Santa Fe, New Mexico, 1988

17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
FIELD	GROUP	SUB-GROUP	

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

Langmuir-Blodgett films have been made with 3-n-hexadecylpyrrole and 3-n-octadecylpyrrole monomers and copolymers with unsubstituted pyrrole made by chemical polymerization at the air-water interface on a subphase containing $FeCl_3$. Langmuir-Blodgett films consisting of mixtures of stearic acid and alkylsubstituted polythiophenes have also been made as bilayer films. The orientation of single and multilayer films on platinum substrates have been studied by Near Edge X-ray Absorption Fine Structure Spectroscopy which also gives information about charge transfer interactions between the aromatic groups and the metallic substrates. The alkylsubstituted pyrroles form highly ordered two-dimensional structures. $FeCl_3$ initiated copolymerization with unsubstituted pyrrole leads to a more disordered system. In the case of polythiophene-stearic acid bilayers, the stearic acid layers are highly ordered. The poly(alkyl thiophene) layers sandwiched between stearic acid layers, on the other hand, exhibit random orientation of the thiophene moieties.

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PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT ACCESSION NO.

11. TITLE (Include Security Classification)

A FTIR and NEXAFS Study of Polypyrrole Langmuir-Blodgett Films

12. PERSONAL AUTHOR(S) X.Q. Yang, J. Chen, P.D. Hale, T. Inagaki, T.A. Skotheim, Y. Okamoto, L. Samuelson, S.K. Tripathy, K. Hong, M.F. Rubner and M.L. DenBoer

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SUPPLEMENTARY NOTATION: Synthetic Metals 28 C251-256 (1989), Proceedings of ICSM conference, Santa Fe, New Mexico, 1988.

17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
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19. ABSTRACT (Continue on reverse if necessary and identify by block number)

3-n-hexadecylpyrrole and 3-n-octadecylpyrrole have been synthesized, and Langmuir Blodgett films of these compounds have been studied by means of NEXAFS and FTIR spectroscopy. By comparing the spectra of two different polarizations, it was found that the hydrocarbon chains are normal to the substrate surface for LB films with multi-layers. For the monolayer LB films, the hydrocarbon tails are tilted towards the substrate surface due to the interaction between the pyrrole ring and the platinum substrate.

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PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT ACCESSION NO

11. TITLE (Include Security Classification)

Chemically Modified Polypyrrole

12. PERSONAL AUTHOR(S) T. Inagaki, T.A. Skotheim, H.S. Lee, Y. Okamoto L. Samuelson and S.K. Tripathy

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15. PAGE COUNT
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16. SUPPLEMENTARY NOTATION Synthetic Metals 28 C245-C250 (1989), Proceedings of ICSM conference, Santa Fe, New Mexico, 1988.

17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
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19. ABSTRACT (Continue on reverse if necessary and identify by block number)

Polypyrrole (PPy) films have been systematically modified with electroactive groups in the β-position to design electrode materials with specific electrochemical and surface active properties.

Electrochemical copolymerization of pyrrole and 3-(6-ferrocenyl,6-hydroxyhexyl)pyrrole (P-6-Fc) yields a ferrocene functionalized polypyrrole with a controlled amount of ferrocene functionalization. And also, copolymers of pyrrole and 3-(4-(2,5-dimethoxyphenyl)butyl)pyrrole (P-MP) can be made by electrochemical polymerization and converted to the copolymers containing pH dependent electro-active hydroquinone moieties. Derivatized pyrroles have also been incorporated into Langmuir-Blodgett film structures. The surface pressure-area isotherms of 3-(13-ferrocenyl,13-hydroxytridecyl) pyrrole (P-13-Fc) and the mixed monolayer of P-13-Fc and 3-n-hexadecylpyrrole (HDP) are shown.

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PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT ACCESSION NO

11. TITLE (Include Security Classification)
Monolayer Formation Characteristics of Novel Organic Molecules with Nonlinear Optically Active Moieties

12. PERSONAL AUTHOR(S) S.S. Kumar, R.S. Kumar, L.A. Samuelson, J. Kumar, A. Blumstein and S.K. Tripathy

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15. PAGE COUNT
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16. SUPPLEMENTARY NOTATION to be published in Thin Solid Films, Proceedings of the Fourth International Conference On Langmuir-Blodgett Films, April 24-29, 1989, Tsukuba, Japan

17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
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19. ABSTRACT (Continue on reverse if necessary and identify by block number)

A number of novel electroactive materials with amphiphilic molecular structure containing COOH, OH, NMe_2, OMe, NO_2 and CN were synthesized. This paper discusses the monolayer forming ability of these novel compounds. The stability of the monolayer as a function of pH, temperature, composition of the material in mixed monolayers and the nature of the subphase are investigated. Appropriate choice of experimental conditions under which a stable monolayer is obtained for each material is indicated.

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11. TITLE (Include Security Classification)
Cognition and Order in Langmuir-Blodgett Films of a 3-Hexadecyl Pyrrole and Ferrocene-Derivatized Pyrrole Mixed Monolayer System

12. PERSONAL AUTHOR(S) L. Samuelson, A.K.M. Rahman, G.P. Puglia, S. Clough, S. Tripathy, T. Inagaki, X.Q. Yang, T.A. Skotheim, Y. Okamoto

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15. PAGE COUNT
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16. SUPPLEMENTARY NOTATION Proceedings of International Workshop On Intelligent Materials, The Society of Non-Traditional Technology, Tsukuba Center for Institutes, Japan, March 15-17, 1989, to be published in Thin Solid Films

17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
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19. ABSTRACT (Continue on reverse if necessary and identify by block number)

Novel, self-assembled materials have been designed and produced from first principle to possess unique structural hierarchy and electronic and optical properties. The Langmuir-Blodgett technique was used to study the molecular organization of a mixed 3-hexadecyl pyrrole (3HDP) and ferrocene-derivatized pyrrole (Fc-Py) surfactant system. The pyrrole moiety was chosen for its' well established electronic and optical properties when polymerized, while ferrocene, it is theorized, if properly oriented into a Langmuir-Blodgett monolayer film may show a layered array of transition metals which would be extremely valuable as a model for two-dimensional magnets. The ferrocene group may also provide the possibility of charge coupling between neutral ferrocene and oxidized ferricenium which could be controlled electrochemically or photochemically. The combination of these two moieties in a highly ordered molecular superlattice coupled with the ability to control the growth technique electrochemically, photochemically or by self-assembly should provide an effective means of directly controlling the material's (continued)

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11. TITLE (Include Security Classification)

Nonlinear Optics and Organic Materials

12. PERSONAL AUTHOR(S) Elizabeth Cavicchi, Jayant Kumar, Sai Kumar, Sukant Tripathy

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15. PAGE COUNT
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16. SUPPLEMENTARY NOTATION

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17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
FIELD	GROUP	SUB-GROUP	

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

The origin and nature of nonlinear optical effects in organic and polymeric materials is discussed. Approaches to the applications of these nonlinear optical effects in prototypical devices is explored.

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